1948

Microchemical identification of Amidone.

Robert C. Watson
University of Louisville

UNIVERSITY OF LOUISVILLE

MICROCHEMICAL IDENTIFICATION OF AMIDONE

A Dissertation

Submitted to the Faculty

Of the Graduate School of the University of Louisville

In Partial Fulfillment of the

Requirements for the Degree

Of Master of Science

Department of Chemistry

By

Robert C. Watson

Year

1948

NAME OF STUDENT: Robert C. Watson

TITLE OF THESIS: Microchemical Identification of Limestone

APPROVED BY READING COMMITTEE COMPOSED OF THE FOLLOWING MEMBERS:

M. I. Bowman

G. L. Corley

A. E. Harvey, Jr.

NAME OF DIRECTOR: M. I. Bowman

DATE: Oct. 9 1948

Acknowledgements

The writer takes this opportunity to acknowledge his indebtedness to Raymond A. Bevins, Identification Section, Alcohol Tax Unit, United States Treasury Department, for his assistance in making the photomicrographs and for preparing the plates used in this paper.

He also wishes to express his gratitude to Dr. Max I. Bowman, Assistant Professor of Chemistry, College of Arts and Sciences, University of Louisville, for his council and direction, and for his helpful criticism of the manuscript.

Contents

Purpose

The purpose of the experimental work dealt with in this paper was to develope microchemical methods and tests to be used in the identification of Amidone.

At the present time there is in the literature little or no reference to suitable methods of identification of this new drug. Since Amidone is classed as a dangerous drug and placed under the Harrison Narcotic Act, it is highly desirable to have identifying tests to detect the presence of Amidone in seizures of illicit drugs by the Bureau of Narcotics.

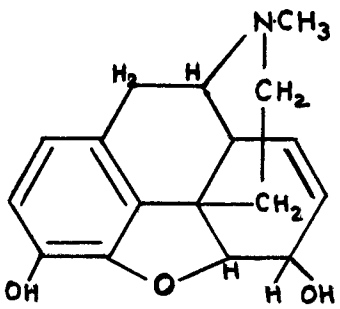
Historical

In the 1930's the chemists in Germany were looking for an antispasmodic as a substitute for atropine and developed a compound which they called Dolantin. It was discovered that this drug was a better analgesic than antispasmodic and they introduced it to medicine as such in 1939. Subsequently Dolantin (known in this country as the drug Demerol) was shown to possess many morphine-like properties including those of tolerance and addiction liability, but its analgesic potency was more nearly like codeine than morphine.

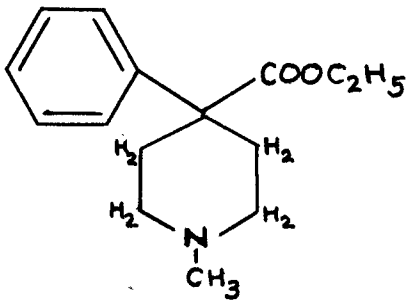
The Germans made many Demerol derivatives, but apparently none showed more than minor improvement. At the same time they were developing a series of diphenyl derivatives and some of these showed remarkable activity. The outstanding example of the series the Germans called "10820" or "Amidon". Accounts of this substance have appeared in American literature under these designations and as "Dolophine" and "AN-148". Methadon is the name recommended for it by the Council on Pharmacy and Chemistry of the American Medical Association. Amidone is the name

which has been most commonly employed in investigative work.

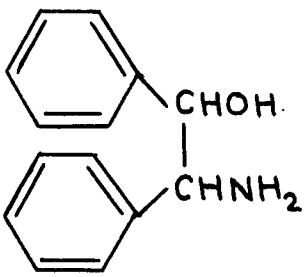
It should be emphasized, however, that all of the names mentioned refer to the same chemical entity. Amidone then is 6-dimethylamino-4,4-diphenyl-3-heptanone. Chemically it is not at all related to morphine nor directly to Demerol. It is somewhat related to a diphenyl ethanolamine which has been described as mildly analgesic, but which is not at all morphine-like. The structural formulas for these compounds are shown below.



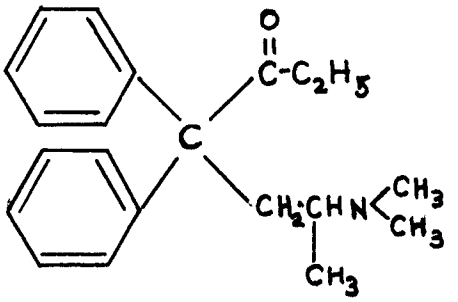
MORPHINE



DEMEROL



B-HYDROXY-α,b-DIPHENYL ETHYLAMINE



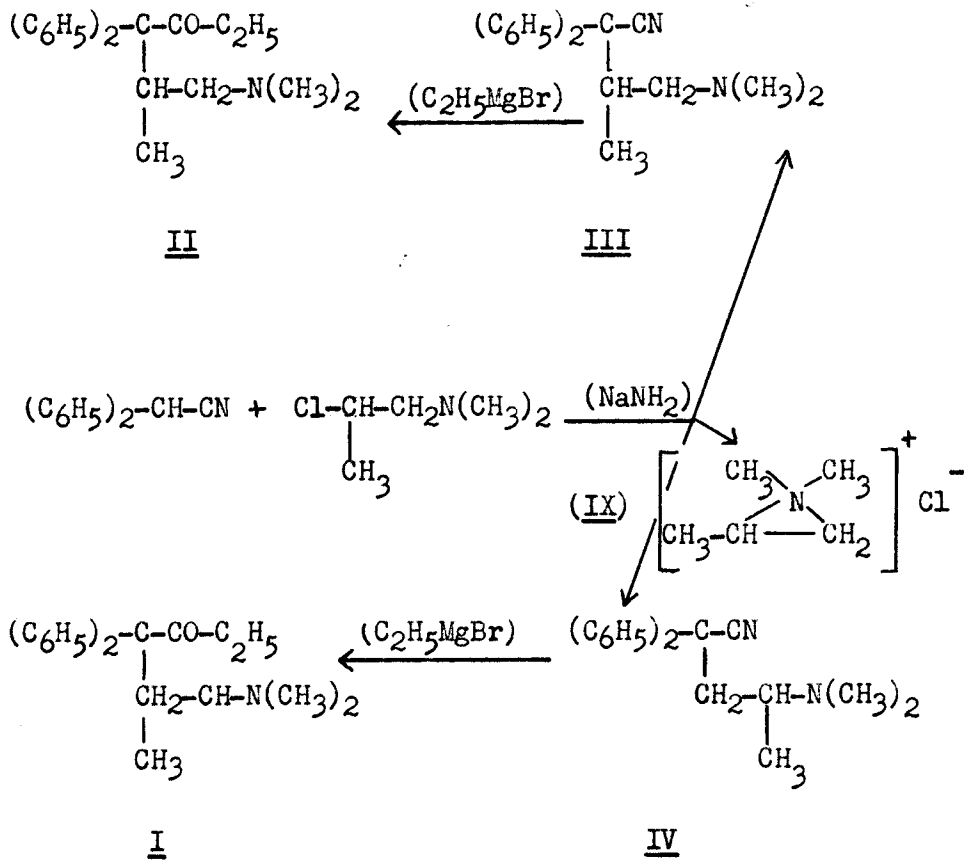
AMIDONE

The hydrochloride of Amidone is a white crystalline substance, having a melting point of 236° C. It is soluble in water to about 5%, has a bitter taste, and is somewhat irritating when injected subcutaneously. Amidone is a racemate and has been resolved into its levo- and dextrorotatory isomers.

The racemate and the isomers have about the same toxicity in laboratory animals, but the analgesic effect and other morphine-like properties are exhibited chiefly by the l-form. In animals Amidone is three to ten times more toxic than morphine, according to the species, and two to three times more toxic than Demerol, but its analgesic effect is twice that of morphine and ten times greater than that of Demerol so it still has a very wide margin of safety.(1)

Using the German procedure in preparing Amidone, the Medical Research Division of Sharp and Dohme, Inc., led to the finding that the reaction between diphenylacetonitrile and 1-dimethylamino-2-chloropropane results in a mixture containing equal amounts of two isomeric aminonitriles, III (m.p. 66-67°) and IV (m.p. 90-91°). The high-melting nitrile (IV) reacted with ethylmagnesium bromide to

yield a product that possessed all of the properties reported for Amidone.



From these data, it appears that Amidone possesses the structure I although the structure II should result if the reaction followed a normal course. It seems probable that the reaction proceeds through the ethyleneimmonium ion (IX) in a manner similar to that demonstrated for other reactions of the halogenated alkylamines.(3)

The toxic doses of the optical isomers of Amidone upon intravenous injection in mice all lie between 15 and 18 mg./Kg. for LD50 value; for iso-Amidone nitrate the comparative figure is approximately 40 mg./Kg. (6)

The Approach to the Problem

1. All available compounds and reagents known to give crystalline precipitates with plant alkaloids, synthetic drugs, etc., were first tested with a 1/50 solution of Amidone for crystals.

2. From an inspection of the chemical formula of Amidone it was predicted that the ketone group would be most active. Therefore reactions with sodium bisulfite, hydroxylamine, phenylhydrazine, semicarbazide were tried. None of these reagents gave crystals with Amidone under the conditions of the experiment.

3. Screening tests were made on all other available elements and compounds and those not giving crystalline precipitates with Amidone were eliminated.

4. Substances giving crystals with Amidone were then further screened and selected by applying four principles of good crystal-forming reagents. (see page 12)

5. Ten substances were finally selected as reagents under step four and the best concentrations were arrived at by experiment.

6. The salts of all available alkaloids and synthetic

drugs were tested with each of the ten reagents to determine if similar crystals were formed with Amidone and to select the reagents giving the fewest crystalline precipitates with the common alkaloids.

7. Photomicrographs were made of the characteristic crystal formation with Amidone and the various reagents. Photographs were also made of the crystals formed by the selected reagents and the common alkaloids.

Experimental

Neutral aqueous solutions were made of all substances tested. For the most part 1% solutions were prepared, however, saturated solutions were made of compounds having low water solubility. Mild heat was applied when necessary to effect a clear solution. All solutions were filtered before testing. Water insoluble compounds were tested for solubility in dilute HCl and NH_4OH.

In general the same procedure was followed in making all of the preliminary screening tests. Three separate drops of the test solution were placed on a clean glass slide. One drop was acidified with dilute HCl, another made basic with dilute NH_4OH while the third was left neutral. If a precipitate formed after the addition of the acid or base the drop was removed from the slide and not futher tested. Next a drop of Amidone solution (1/50) was placed on the slide adjacent to each test drop and allowed to flow into it. Microscopic examination of the slide was made at once, using the low power objective. If there was no immediate crystal formation the slide was allowed to stand for fifteen minutes and examined again. If no crystals had formed on standing the

material on the slide was discarded. Although all slides were examined under ultra-violet light, none showed fluorescent activity.

Substances Giving Crystals With Amidone

Of 197 substances tested only 42 gave crystalline precipitates with Amidone under the conditions of the experiment. These are shown in the following table. Unless otherwise indicated the concentration of the Amidone solution was 1:50 and the test solutions 5%.

Test Solution	Crystal Formation				
	Class* A	Class B	Class C	Class D	Class E
Ammonium Acetate		x	x		
Ammonium Carbonate	x	x			
Ammonium Hydroxide	x	x			
Ammonium Iodide				x	x
Ammonical Nickel Acetate (2)		x	x		
Ammonium Oxalate		x	x		
Ammonium Sulfide	x			x	
Ammonical Silver Nitrate (2)		x	x		
Barium Acetate	x		x	x	
Barium Chloride		x	x		
Barium Hydroxide (sat.soln.)		x	x		x
Bromine Water (sat.soln.)				x	x
Cadmium Iodide		x			x
Calcium Chloride	x	x	x		
Hydrobromic Acid (conc.)	x	x			
Hydriodic Acid (47%)	x			x	
Iodine (sat. soln.)				x	x

(Continued to next page)

Substances Giving Crystals With Amidone (Concluded)

Test Solution	Crystal Formation				
	Class* A	Class B	Class C	Class D	Class E
Mercuric Chloride				x	x
Mercuric Nitrate (sat.soln.)				x	x
Magnesia Mixture (2)	x		x	x	
Nickel Acetate		x	x		
Palladium Chloride (in dilute HCl)				x	x
Potassium Acetate		x	x		
Potassium-Bismuth Iodide	x	x	x		
Potassium Bitartrate		x	x		
Potassium Chromate	x			x	
Potassium Ferrocyanide				x	x
Potassium Iodide				x	x
Potassium Nitrate		x	x		
Potassium Oxalate		x	x		
Saccharin	x			x	
Silver Iodide-Potassium Iodide (1:15)	x	x			
Sodium Acetate			x	x	
Sodium Bicarbonate	x	x			
Sodium Borate	x	x			
Sodium Carbonate	x	x			
Sodium Fluoride		x	x		
Sodium Iodide			x	x	
Sodium Phosphate (dibasic)	x	x	x		
Sodium Silicate (sat. soln.)	x	x	x		
Sodium Sulfite	x	x	x		
Sodium Tungstate		x	x		

*Class A - Crystal shape not definite or distinct.
Class B - Crystals slow in forming.
Class C - Crystals probably due to reagent being thrown out of solution.
Class D - Rapid crystal formation.
Class E - Crystals having definite and distinctive shapes.

Selection Of Reagents

Ten substances were selected as having the most possibilities for reagents. This selection was based on their appearance under Class D and Class E of the preceeding table. These compounds were reexamined and arranged below in order of their decreasing desirability.

Test Solution	Qualities Possessed for Good Crystal Formation			
	Criteria I*	Criteria II	Criteria III	Criteria IV
1. 1% Palladium Chloride in dilute HCl.....	x	x	x	x
2. 5% Potassium Ferrocyanide (freshly made).....	x	x	x	
3. Dilute Bromine Water.....	x	x	x	
4. Saturated Iodine Water.....		x		x
5. 1% Potassium Iodide.....		x	x	
6. 3% Sodium Iodide.....		x	x	
7. 1% Mercuric Nitrate.....			x	
8. Iodine-Potassium Iodide (dilute solution).....				x
9. 5% Cadmium Iodide.....			x	
10. 1% Mercuric Chloride.....			x	x

*Criteria I. Crystals should form rapidly from widely varying concentrations of sample and reagent, and should have well defined and characteristic shapes.

Criteria II. Slight variations in pH of media should not affect shape, formation time, etc., of crystals, nor should the presence of small amounts of impurities interfere with the test.

The proper concentration of reagents were determined by testing a 1/200 solution of Amidone with 1, 2, 5, 10 and 20% solutions of each reagent. After this was done each reagent was tested with Amidone solutions of the following concentrations: 1/50; 1/200; 1/500; and 1/1000. This was done to establish the optimum concentrations for each reagent and to ascertain the limit of reaction for each test. Photomicrographs were made of characteristic crystals formed with each reagent. (See Plates I to VI.)

The salts of 35 available narcotic alkaloids and synthetic drugs were tested with the selected reagents. (See Chart I.) Photomicrographs were made of the crystals produced with the more common alkaloids. (See Plates I to VI.)

Criteria III. A minimum of crystalline precipitates should be given with other drugs and these crystals should have distinctive shapes.

Criteria IV. The reagent should have a maximum of stability and should not be precipitated itself in a crystalline form resembling the crystals formed with the compound being examined.

Substances Not Giving Crystalline Precipitates With Amidone

Under the conditions of the experiment the following substances did not yield crystalline precipitates with Amidone:

Acetic Anhydride
Acetic Acid
Aluminum-Potassium Sulfate
2-Amino-Sulfuric Acid
Ammonium Chloride
Ammonium Molybdate
Ammonium Thiocyanate
Ammonium Vanadate
Antimony-Potassium-Tartrate
Antimony Sulfide
Antimony tri-Chloride
Arsenic Chloride
Arsenic Trioxide
Barium Nitrate
Benzidine
Benzoic Acid (reagent crystals)
Betanaphthal
Bismuth Nitrate
Boric Acid
Cadmium Chloride
Cadmium Sulfide
Chloral Hydrate
Calcium Carbonate
Calcium Hydroxide
Calcium Sulfate
Carbolic Acid
Citric Acid
Chromic Acid
Chromium Trichloride
Chromium Nitrate
Cobalt Acetate
Cobalt Chloride
Cobalt Nitrate
Cobalt Sulfate

Cobalt Thiocyanate (blue ppt.)
Cupric Acetate
Cupric Ammonium Chloride
Cupric Bromide
Cupric Carbonate
Cupric Chloride
Cupric Nitrate
Cupric Sulfate
p-Dimethyl Aminobenzaldehyde
Dimethyl Glyoxime
2-4-Dinitro-phenylhydrazine
Diphenyl
Diphenylamine
Ferric Chloride
Ferric Ammonium Citrate
Ferric Ammonium Sulfate
Ferric Nitrate
Ferrous Chloride
Ferrous Sulfate
Formic Acid
Frohedes Reagent (4)
Gallic Acid
Hydrochloric Acid
Hydroxylamine-HCl
Iodic Acid
Iridium Chloride
Krauts Reagent (4)
Lactic Acid
Lanthanum Nitrate
Lead Acetate
Lead Iodide
Lead Subacetate
Lithium Carbonate
Lithium Chloride
Lithium Nitrate
Magnesium Chloride
Marme's Reagent (4)
Mayer's Reagent (4)
Manganous Chloride
Mercuric-Potassium-Iodide
Millons Reagent (4)
Molybdic Acid
Nickel Chloride
Nickel Nitrate

Nickel Sulfate
Nitric Acid
o-Nitrobenzaldehyde
Oxalic Acid
Picric Acid
Picrolonic Acid
m-Phenylene-diamine-HCl
n-Phenylene-diamine-HCl
Phenylhydrazine-HCl
Phloroglucinol
Phosphomolybdic Acid
Phosphoric Acid
Phospho-Tungstic Acid
Phthalic Anhydride
Platnium Chloride
Potassium Acid Phthalate
Potassium Arsenate
Potassium Bromate
Potassium Bromide
Potassium Bisulfide
Potassium Carbonate
Potassium Chlorate
Potassium Chloride
Potassium Cyanide
Potassium Dichromate
Potassium Ferricyanide
Potassium Hydroxide
Potassium Iodate
Potassium Permanganate
Potassium Phosphate
Potassium Sulfide
Potassium-Zinc-Iodide
Pyrogallic Acid
Resorcinol
Rhodanine
Rhodium Chloride
Ruthenium Chloride
Salicylous Acid
Selenious Acid
Semi-Carbazide-HCl
Silico-Tungstic Acid
Silver Acetate
Silver Sulfate
Sodium Benzene-Sulfonate

Sodium Benzoate

Sodium Bichromate

Sodium Bisulfite

Sodium Chloride

Sodium-Cobalt-Nitrate

Sodium Formate

Sodium meta-Bisulfite

Sodium Nitrate

Sodium Nitrite

Sodium Nitro Prusside

Sodium Peroxide

Sodium Phospho-Molybdate

Sodium Sulfate

Sodium Sulfide

Sodium Thiocyanate

Sodium Tartrate

Sodium Thiosulfate

Sulfamic Acid

Sulfuric Acid

Sulfurous Acid

Stannous Chloride

Strontium Chloride

Tannic Acid

Tartaric Acid

Tetrachloro-Phthalic Acid

Tetrachloro-Phthalic Anhydride

Uranium Acetate

Uranium Nitrate

Wagner's Reagent (4)

Zinc Acetate

Zinc Chloride

Zinc-Chloro-Iodine

Zirconium Nitrate

Detailed Description of Microcrystalline Tests With Amidone

Palladium Chloride - These crystals form almost at once from an amorphous precipitate in the 1/50 solution and somewhat slower in the 1/200 solution. In greater dilutions the crystals were very slow in forming and resembled double headed flint spearheads. These crystals show brilliantly under polarized light. They grow to considerable size, forming rosettes of brown fern-like plates or fragments. One notable feature is the fact that the rod-like crystal growth occurs on one side only of each arm and at an angle of approximately 30° from the main axis. This was found to be the most satisfactory crystalline test. (Plate I, Figs. 1, 2, 3, 4)

Potassium Ferrocyanide - Crystals form at once from a clear solution in the 1/50 concentration. At first these crystals appear as bundles of needle-like rods curving outward at the ends, having somewhat the appearance of sheaves of grain. Later rosettes of plates were formed. Crystals were not formed in the more dilute solutions. The three dimentional growth of these crystals may best be observed under polarized light. (Plate II, Figs. 3, 4, 5)

Bromine Water - Many small crystals formed at once in all solutions, but those in the 1/500 and 1/1000 solutions were best for identification purposes. This test was improved by adding a drop of dilute HCl to the Amidone solution and stirring before adding the bromine water. The crystals at first appear to be single plates having notched ends in such a manner as to resemble the letter "N", when slightly out of focus. Later they develop into rosettes of rod-like plates. (Plate III, Fig. 1)

Iodine Water - Small plates, having notched ends similar to those produced with bromine water, first appear in the 1/50 solution of Amidone. In the more dilute solutions few crystals were formed. These crystals were slow in forming and small in size. Crystal formation may be improved by the addition of a drop of dilute HCl to the sample drop. (Plate III, Fig. 2)

Potassium Iodide - Heavy rosettes of fern-like crystals formed at once in the 1/50 solution of Amidone. The more dilute solutions on standing gave a few small crystals. In the 1/50 solution the crystals started as dark shapeless masses

which later began to branch. The crystal form was very dense and lacked detail, but had a characteristic shape. (Plate III, Fig. 3)

Sodium Iodide - These crystals have the same general appearance as those formed with potassium iodide. The addition of a drop of dilute HCl to the sample drop causes clearer crystal formation.

Mercuric Nitrate - These crystals form best in a neutral solution. A precipitate was formed in the 1/50 solution of Amidone only, from which crystals formed at once. These crystals sometimes dissolved in the reagent drop upon stirring. The crystals formed as rosettes of needles. This is not a sensitive test. (Plate III, Fig. 5)

Iodine-Potassium Iodide - Crystals formed rapidly in all solutions. The crystalline form was affected greatly by the concentration of the sample. In the 1/50 solution the crystals were rectangular plates while in the 1/500 solution they showed three dimentional growth and frequently resembled the letters "H", "Y", and "X". (Plate IV, Figs. 5, 6)

Cadmium Iodide - Rosettes of root-like crystals formed slowly from the 1/100 solution of Amidone. Their growth was three dimentional and only a portion of the crystal was in focus at one time. Crystals formed in the 1/200 solution were brown in color and not clearly defined. (Plate III, Fig. 6)

Mercuric Chloride - The best crystals occured in the 1/200 solution of Amidone. They appeared at once from an amorphous precipitate as rosettes and bundles of rods which may be viewed to advantage under polarized light. (Plate VI, Fig. 1)

Amidone, when allowed to crystallize from a saturated aqueous solution, first appears as diamond shaped plates.

Preparation of Reagents For Microcrystalline Tests

1. Palladium Chloride - (Approximately 1% in dilute HCl). Make up 1 gram $PdCl_2 \cdot 2H_2O$ to 100 ml. with dilute HCl (35 ml HCl s.g. 1.19 q.s. 100 ml with distilled water), warm gently until solution is complete.

2. Potassium Ferrocyanide - (Approximately 5% aqueous solution). Make up 5 grams $K_4Fe(CN)_6 \cdot 3H_2O$ to 100 ml with distilled water. (4) This reagent should be made up fresh each day since it slowly decomposes on standing.

3. Bromine Water - (Dilute solution). Make up 3.5 ml saturated bromine water to 100 ml with distilled water. This reagent should be made up frequently.

4. Iodine Water - (Saturated solution). Place about 1 gram I_2 in a test tube containing about 15 ml distilled water and warm gently over a flame until the I_2 vapors completely saturate the solution, cool and filter. For best results this solution should be completely saturated.

5. Potassium Iodide - (Approximately 1% solution). Make up 1 gram KI to 100 ml with distilled water. This solution may become yellow in time due to oxidation and should, therefore, be made up frequently.

6. Sodium Iodide - (Approximately 2% solution). Make up 3 grams NaI to 100 ml with distilled water. This solution may become brown in time due to the liberation of iodine and should be made up frequently.

7. Mercuric Nitrate - (Approximately 1% solution). Add 1 gram $Hg(NO_3)_2 \cdot H_2O$ to 100 ml distilled water. An insoluble basic salt is formed in dilute aqueous solutions or upon heating. Filter the solution, using only the clear filtrate in tests.

8. Iodine-Potassium Iodide - (Dilute solution). Make up 1.25 gram KI and 0.25 gram I_2 to 250 ml with distilled water.

9. Cadmium Iodide - (Approximately 5% solution). Make up 5.0 grams CdI_2 to 100 ml with distilled water. This solution may become yellow on long exposure to light and should be, therefore, made up frequently.

10. Mercuric Chloride - (Approximately 1% solution). Make up 1 gram $HgCl_2$ to 100 ml with distilled water.

Notes on Making Photomicrographs

In making photomicrographs it was sometimes found advisable to use a cover glass on the sample drop. In other cases this caused the crystals to become distorted in shape, as well as to increase the formation time.

A 16 mm. low power objective and a No. 10 ocular were used throughout. The camera lens was removed and the microscope draw-tube was extended into the camera bellows, allowing the image to be focused on a ground glass screen. Unusually good negatives were obtained in this manner, but some detail was lost in making the final prints.

Summary and Conclusion

Palladium Chloride (1%) in dilute HCl is a satisfactory reagent for the microcrystalline identification of Amidone, and offers several important advantages over other reagents tested.

Not only does it give characteristic crystals from widely varying solutions but it is not affected by slight differences in pH and gives relatively few crystalline precipitates with other common drugs.

Potassium Ferrocyanide (5%) and dilute Bromine Water make valuable collateral reagents when used in conjunction with Palladium Chloride.

CHART NO. 1

REAGENTS

GIVING CRYSTALLINE PRECIPITATES WITH AMIDONE

Drugs Tested	Palladium Chloride	Potassium Ferrocyanide	Bromine Water	Iodine Water	Potassium Iodide	Sodium Iodide	Mercuric Nitrate	Iodine-Potassium Iodide	Cadmium Iodide	Mercuric Chloride
Dicodid	-	-	-	a	-	-	-	a	a	-
Dilaudid	-	-	-	a	-	-	-	a	a	-
Demerol	-	-	-	a	-	-	-	a	a	-
Cocaine	-*	-	-	a	-	c*	-	a	a	a
Morphine	-	a	-	-	-*	-	a	c	c	-*
Narcotine	a	-	a	a	a	-	a	a	a	-
Codeine	-	-	a	a	-*	c	a	c	c	-
Narceine	-*	c*	-	a	-*	c	a	c	a	a
Thebaine	a	-	-	a	-	-	a	a	a	a
Papaverine	a	a	a	a	a	a	c	c	c	c
Dionin	-	-	-	a	-	-	-	c	a	-*
Pantopon	-	-	-	a	-	-	-	a	c	-
Heroin	-	-	-	-	-	-	-	a	a	-*
Atropine	-	-	-	c	-	-	a	c	a	-
Caffeine	c	-	-	-	-	-	-	-	-	-*
Brucine	c	-	-	c	-*	c	-	c	a	c
Strychnine	c	-*	-	c	c	c	a	c	c	-*
Saccharin	-	-	-	-	-	-	a	-	a	-
Stovaine	-	-	a	a	-	-	a	a	a	a
Quinine	-	a	a	c	-	-	c	a	a	c*
Novocaine	-	-	-	a	-	-	-	a	a	a
Alypin	-	-	a	a	c	c	-	c	a	c
Phenacetin	-	-	-	-	-	-	c	-	-	-
Sulfon-Methane	-	-	-	-	-	-	-	-	-	-

(Continued on next page)

CHART NO. 1

REAGENTS

GIVING CRYSTALLINE PRECIPITATES WITH AMIDONE (Concluded)

Drugs Tested	Palladium Chloride	Potassium Ferrocyanide	Bromine Water	Iodine Water	Potassium Iodine	Sodium Iodide	Mercuric Nitrate	Iodine-Potassium Iodide	Cadmium Iodide	Mercuric Chloride
Sodium Phenobarbital	c	-	-	-	-	-	a	-	-	a
Nembutal	c	-	-	-	-	-	a	-	a	a
Seconal	a	-	-	-	-	-	a	-	a	a
Ephedrine-Amytal	-	-	-	-	-	-	a	-	-	a
Digitalin	-	-	-	-	-	-	-	-	-	-
Nitroglycerine	-	-	-	-	-	-	-	-	-	-
Scopolamine	-	-	-	-	-	-	-	-	-	-
Strophauthin	-	-	-	-	-	-	-	-	-	-
Ergotrate	-	-	-	-	-	-	-	-	-	-
Nicotine	-	-	-	a	-	-	a	a	a	a*

a = Amorphous precipitate
c = Crystalline precipitate

* Disagreement with the findings of Stephenson (5) previously reported. It should be noted that the reagent concentrations discussed in this paper differ in most cases from those used by Stephenson.

CRYSTALS FORMED WITH PALLADIUM CHLORIDE

PLATE I

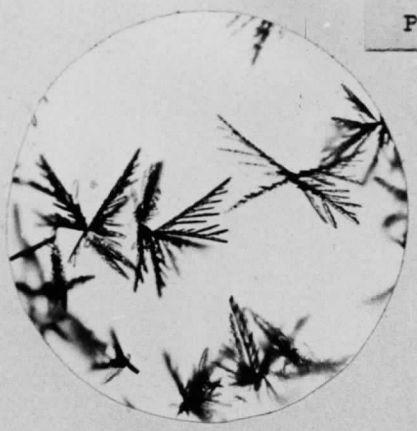


Fig. 1.-Amidone 1 : 50

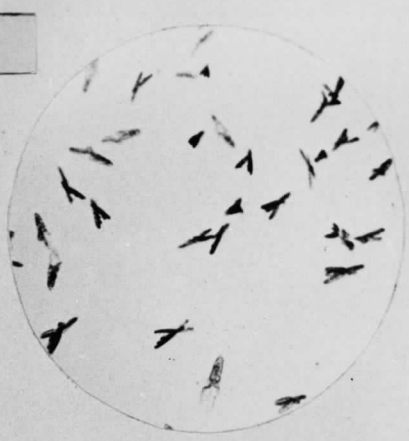


Fig. 2.-Amidone 1 : 200

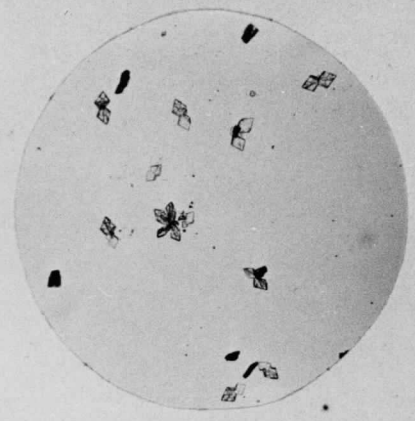


Fig. 3.-Amidone 1 : 500

Fig. 4.-Amidone 1 : 1000

Fig. 5.-Brucine 1 : 100

Fig. 6.-Caffeine 1 : 100

CRYSTALS FORMED WITH PALLADIUM CHLORIDE

PLATE II

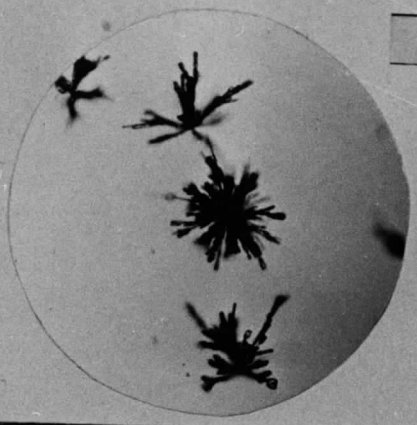


Fig. 1.-Strychnine 1 : 50

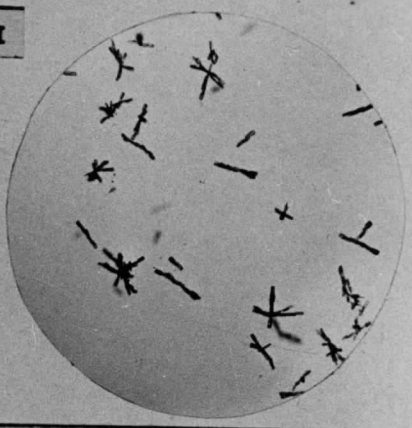


Fig. 2.-Strychnine 1 : 1000

CRYSTALS FORMED WITH POTASSIUM FERROCYANIDE

Fig. 3.-Amidone 1 : 50

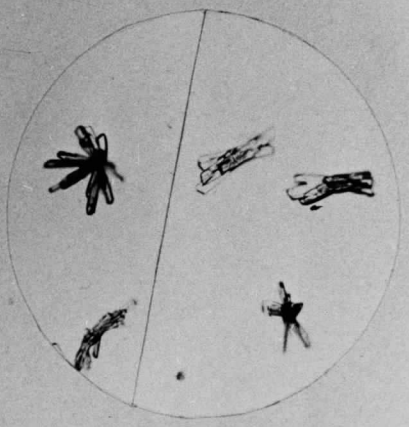


Fig. 4.-Amidone 1 : 50

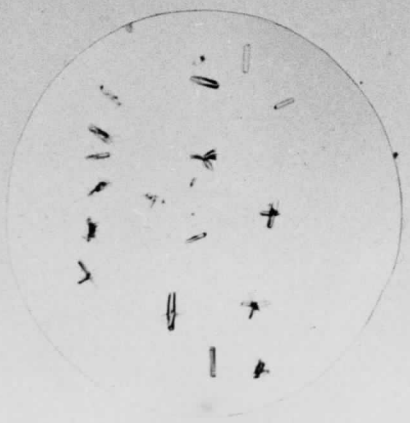


Fig. 5.-Amidone 1 : 200

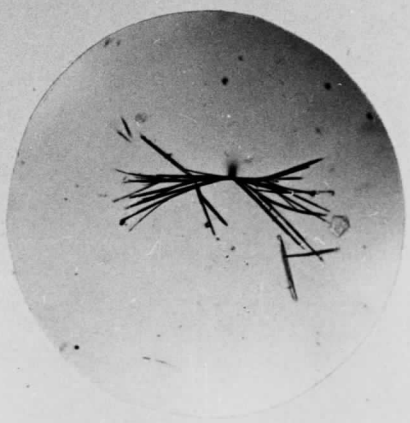


Fig. 6.-Strychnine 1 : 50

CRYSTALS WITH BROMINE WATER

CRYSTALS WITH IODINE WATER

PLATE III

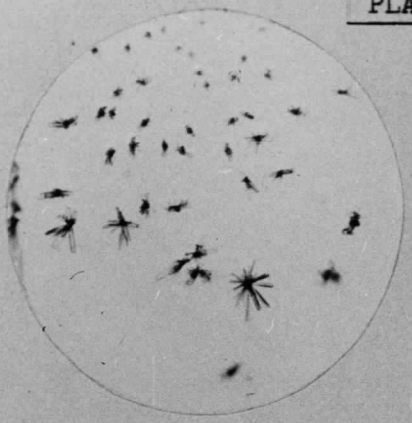


Fig. 1.-Amidone 1:200

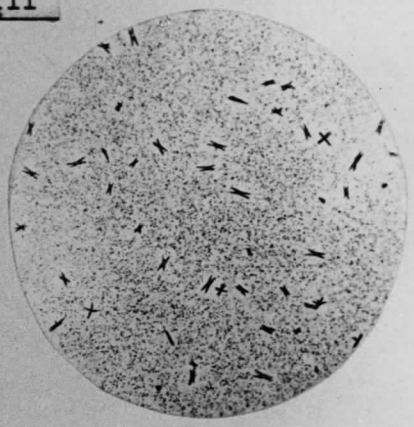


Fig. 2.-Amidone 1:50

CRYSTALS FORMED WITH POTASSIUM IODIDE

Fig. 3.-Amidone 1:50

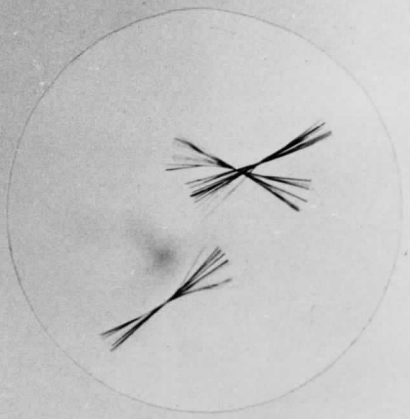


Fig. 4.-Narceine 1:100

CRYSTALS WITH MERCURIC NITRATE

CRYSTALS WITH CADMIUM IODIDE

Fig. 5.- Amidone 1:50

Fig. 6.-Amidone 1:1000

CRYSTALS FORMED WITH SODIUM IODIDE

PLATE IV

Fig. 1.-Amidone 1:50

Fig. 2.-Strychnine 1:50

Fig. 3.-Narceine 1:100

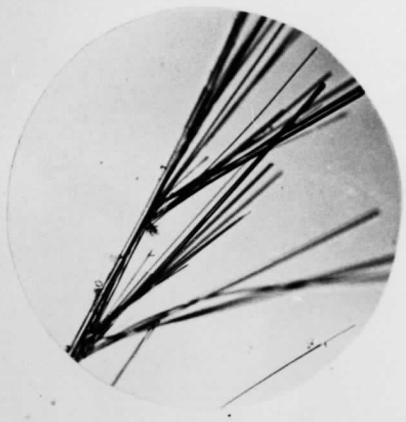


Fig. 4.-Codeine 1:50

CRYSTALS FORMED WITH IODINE-POTASSIUM IODIDE

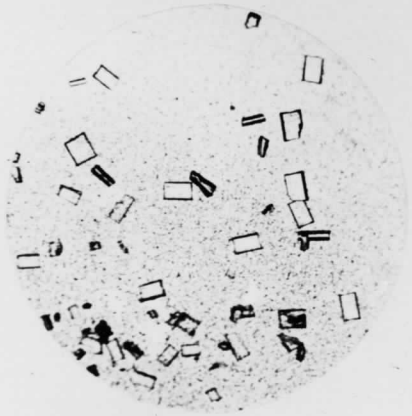


Fig. 5.-Amidone 1:50

Fig. 6.-Amidone 1:500

CRYSTALS FORMED WITH IODINE-POTASSIUM IODIDE

PLATE V

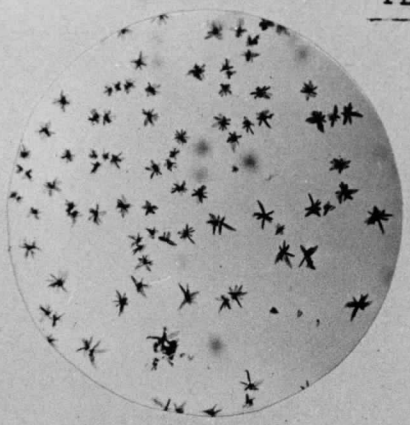


Fig. 1.-Brucine 1:500

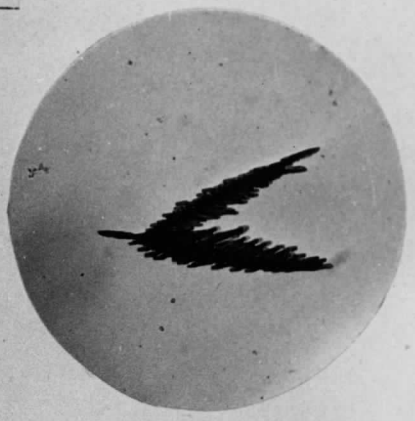


Fig. 2.-Morphine 1:50

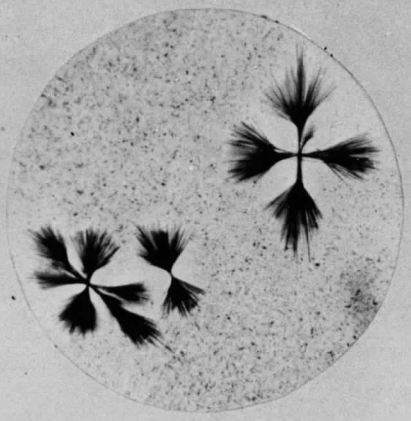


Fig. 3.-Narceine 1:100

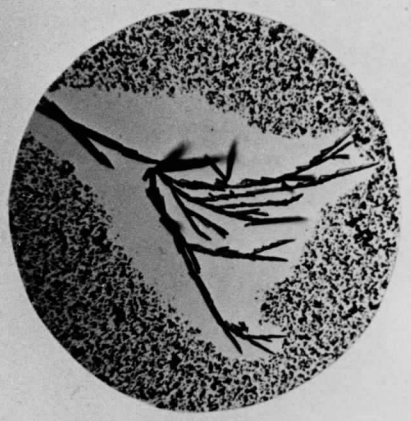


Fig. 4.-Codeine 1:50

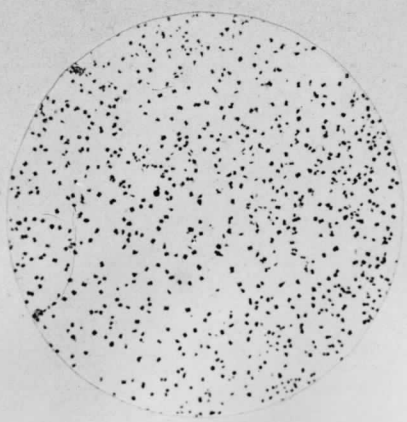


Fig. 5.-Atropine 1:50

CRYSTALS FORMED WITH MERCURIC CHLORIDE

PLATE VI

Fig. 1.-Amidone 1:200

Fig. 2.-Caffeine 1:100

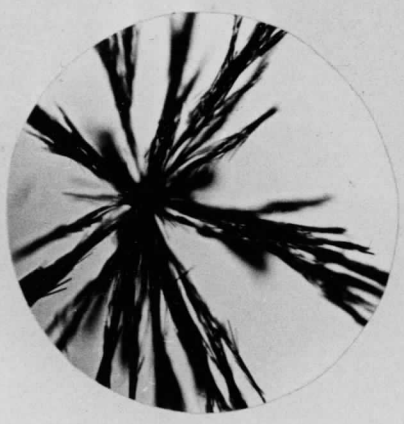


Fig. 3.-Morphine 1:50

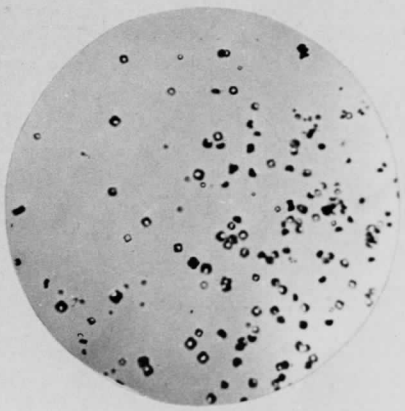


Fig. 4.-Papaverine 1:50

Fig. 5.-Quinine 1:50

Index to Plates

Index to Plates (Concluded)

Literature Cited

(1) Eddy, Nathan B., J. Am. Pharm. Assoc., Prac. Pharm. Ed., 8, 536-40 (1947)

(2) _____, "Methods of Analysis, A. O. A. C.," 6th ed., Association of Official Agricultural Chemists, Washington, D. C., 1945, 739-47.

(3) Schultz, E. M., C. M. Robb, and J. M. Sprague, J. Am. Chem. Soc., 69, 188-9 (1947)

(4) Stephenson, Charles H., "Some Microchemical Tests for Alkaloids," J. B. Lippincott Co., Philadelphia, 1921, 8-10.

(5) Ibid., Table of Reactions following Photographic Plates.

(6) Thorp, R. H., E. Walton and P. Ofner, Nature, 160, 605 (1947)